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✓ Spreadsheet
✓ DTS

MEMORANDUM FOR PRS (In-House)

FROM: PROI (TI) (STINFO) 28 May 1999

SUBJECT: Authorization for Release of Technical Information, Control Number: **AFRL-PR-ED-TP-FY99-0115**
DeRose and Fajardo, "HEDM Source Characterization by Multi-Photon Ionization Time-of-Flight Mass Spectrometry"

Presentation **HEDM Conference** **(Statement A)**

HEDM Source Characterization by Multi-Photon Ionization Time-Of-Flight Mass Spectrometry

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High Energy Density Matter Contractors Conference
Cocoa Beach, FL
8-11 June 1999

NEED A CLEAR STATEMENT OF OBJECTIVE OF THIS PROJECT. EITHER MODIFY ABSTRACT OR ADD AT OBJECTIVE SLIDE. OK

Abstract

Thermochemical calculations show that a High Energy Density Matter (HEDM) fuel consisting of 5% boron atoms in cryogenic solid hydrogen would provide a 21% (or 80 seconds) improvement in Isp over LOX/LH_2.[1] However, to date only about 0.1% metal atom concentration has been achieved in a solid hydrogen matrix. These studies have demonstrated that low concentrations of HEDM species are chemically stable in solid hydrogen. However, in order to obtain higher HEDM species concentrations we must determine optimum matrix deposition conditions. Knowing the dopant species' identities before and after deposition will quantify HEDM species recombination during the deposition process, and will facilitate the desired optimization.

We present results obtained using an apparatus designed to characterize the species produced by a variety of HEDM sources. In this apparatus, the HEDM species are ionized by a pulsed excimer laser beam and analyzed by time-of-flight mass spectrometry. Complications arising from photofragmentation vs. photoionization were encountered and documented as a function of ionization wavelength and intensity.

Data from experiments with HEDM precursor-coated tungsten filament sources are presented. These sources have numerous advantages, including low cost and versatility. Boron atom sources are made by chemical vapor deposition of pyrolized B_2H_6 onto a filament. Characterization of the gas phase products of these sources indicates significant contamination by B_xH_y species.

The time-of-flight apparatus will be used to assist in the development and to verify the operation of future HEDM sources. By comparing what is produced by a source with what is trapped in the matrix, we can obtain a better understanding of the deposition process and insight into obtaining the desired higher concentration HEDM matrices.

1. Carrick, P.G., "Specific Impulse Calculations of High Energy Density Solid Cryogenic Rocket Propellants 1: Atoms in Solid H_2," PL-TR-93-3014, Phillips Laboratory, Edwards Air Force Base, CA, 1993.

Project Objective: To identify species produced by HEDM sources

- Background: HEDM species are chemically stable in H_2 matrices in low concentrations and at low temperatures.

- BUT we want higher concentrations (i.e.,5% HEDM in solid H_2).

- Therefore, knowing the dopant species' identities before and after deposition will result in a better understanding of the deposition process, making it easier to optimize the deposition conditions for high concentration HEDM/solid H_2 matrices.

Sources

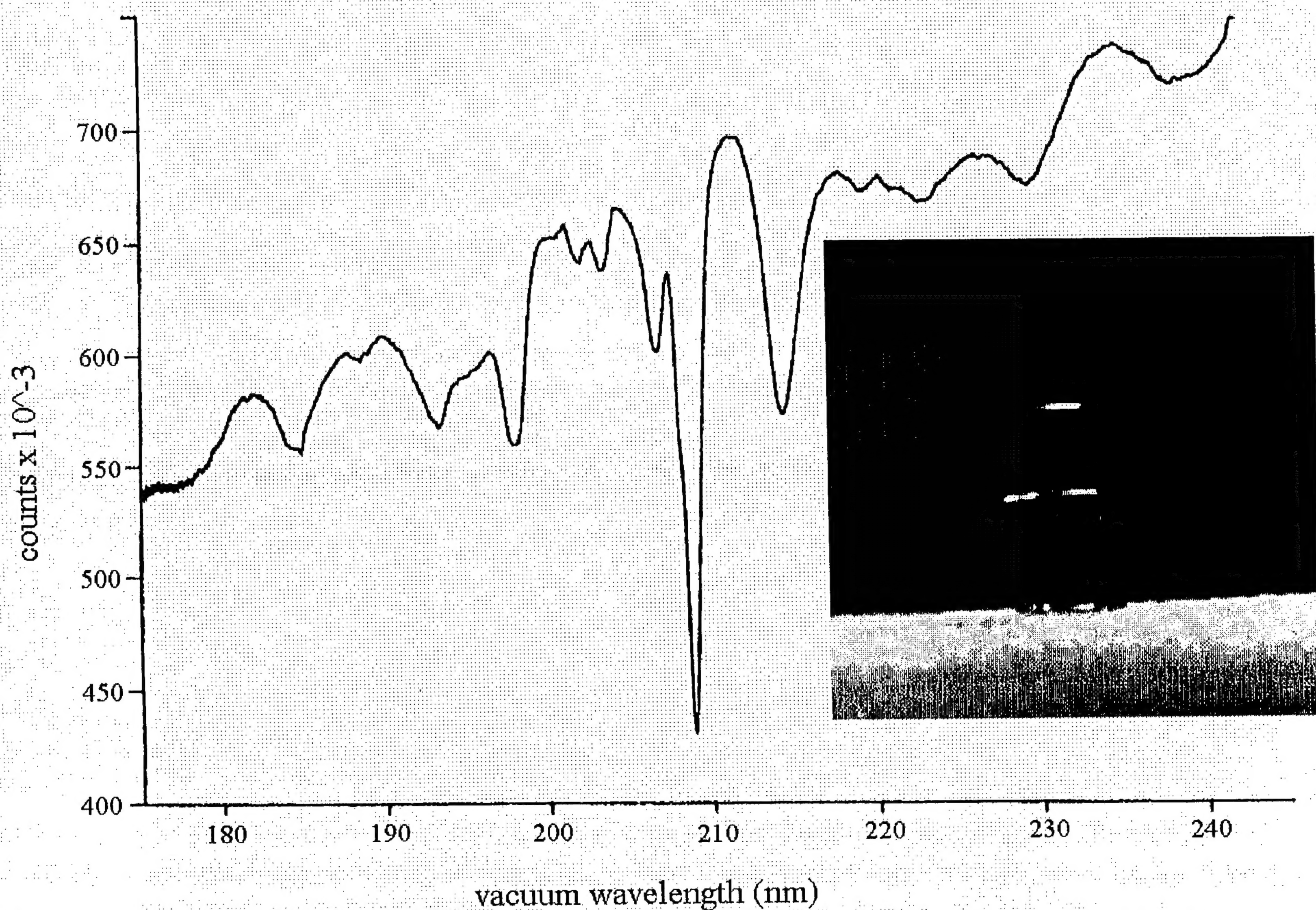
HEDM sources:

- AFRL-developed boron sources
- commercial sources such as Knudsen ovens or electron beam sources
- tungsten filament sources:
  - made by coating the tungsten filament of a quartz halogen light bulb (Figure 1)
  - advantages: inexpensive, easy and fast to make, suitable for numerous species (metals and solids)

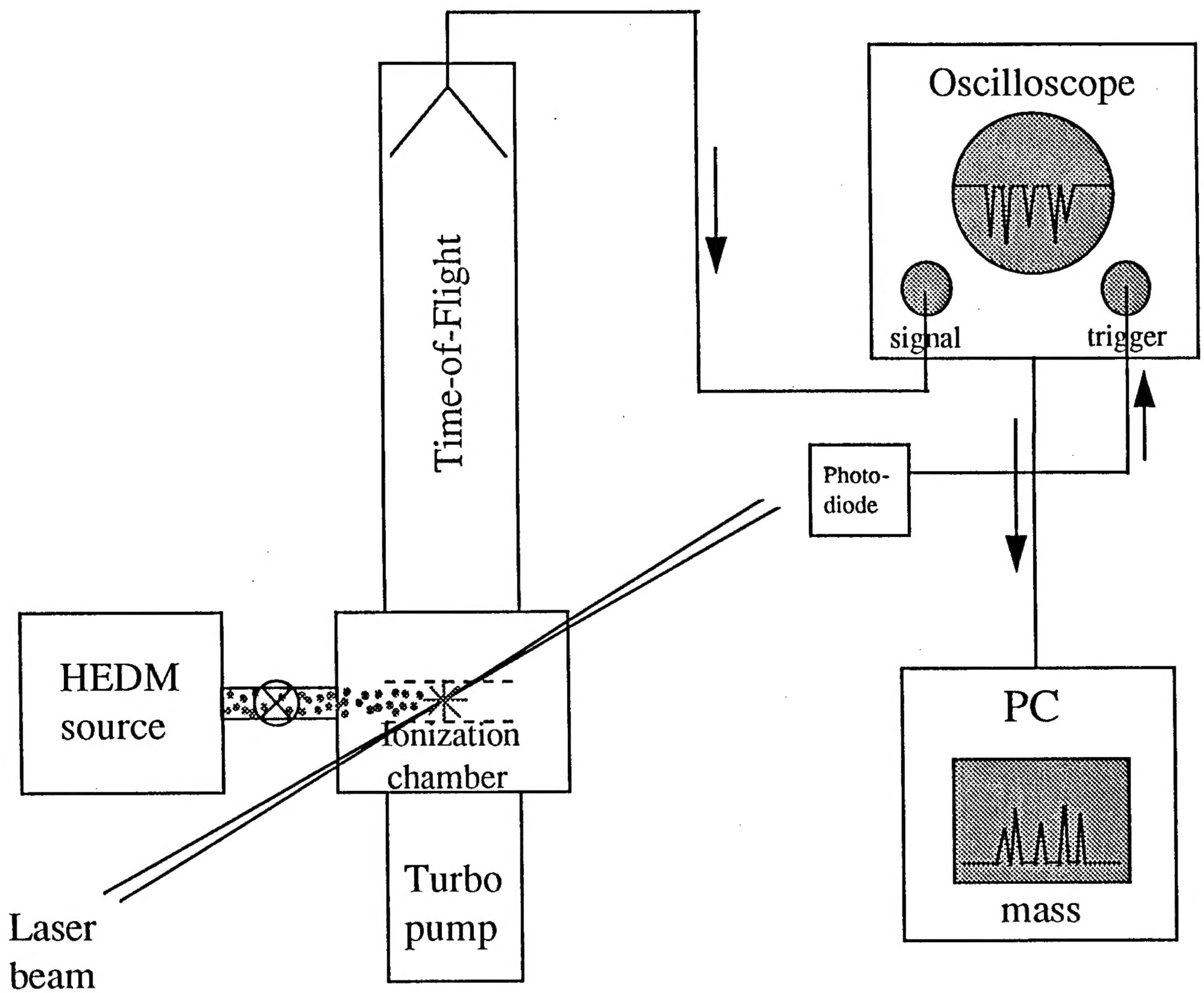
- B sources can easily be made by pyrolizing B_2H_6 onto a bare filament. It has been proven that B sources made by this technique do produce B atoms.

- **However, besides B atoms, other boron-containing species may be produced as well. Or, more generally, any HEDM source may produce many other species besides the target species.**

B atoms in solid argon from B tungsten filament source



Time-of-Flight Mass Spectrometry apparatus for HEDM source characterization



Time-of-Flight Mass Spectrometry apparatus for HEDM source characterization (continued)

1. Source produces beam of HEDM species.

2. HEDM species beam intersects focussed UV laser beam.

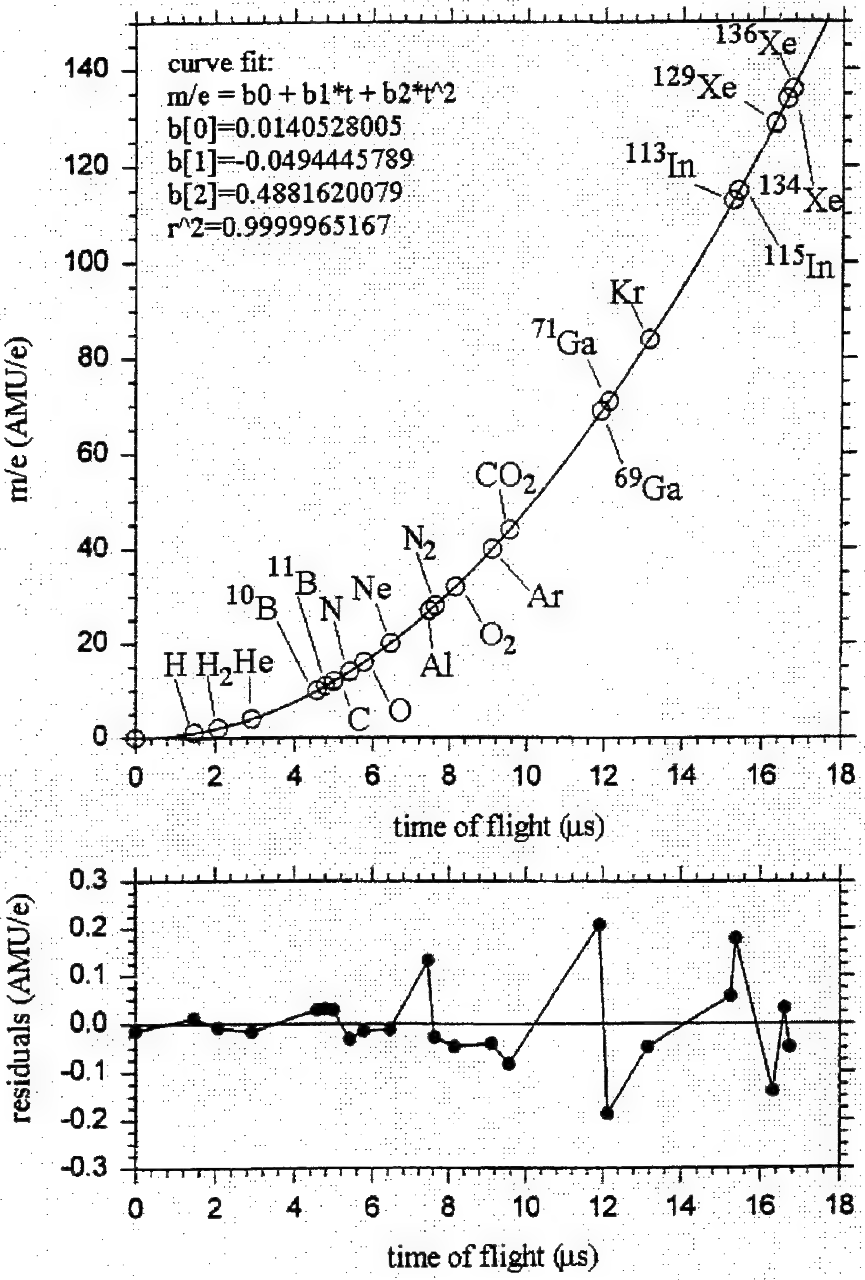
3. MPI and MPF take place at intersection → ION SOURCE

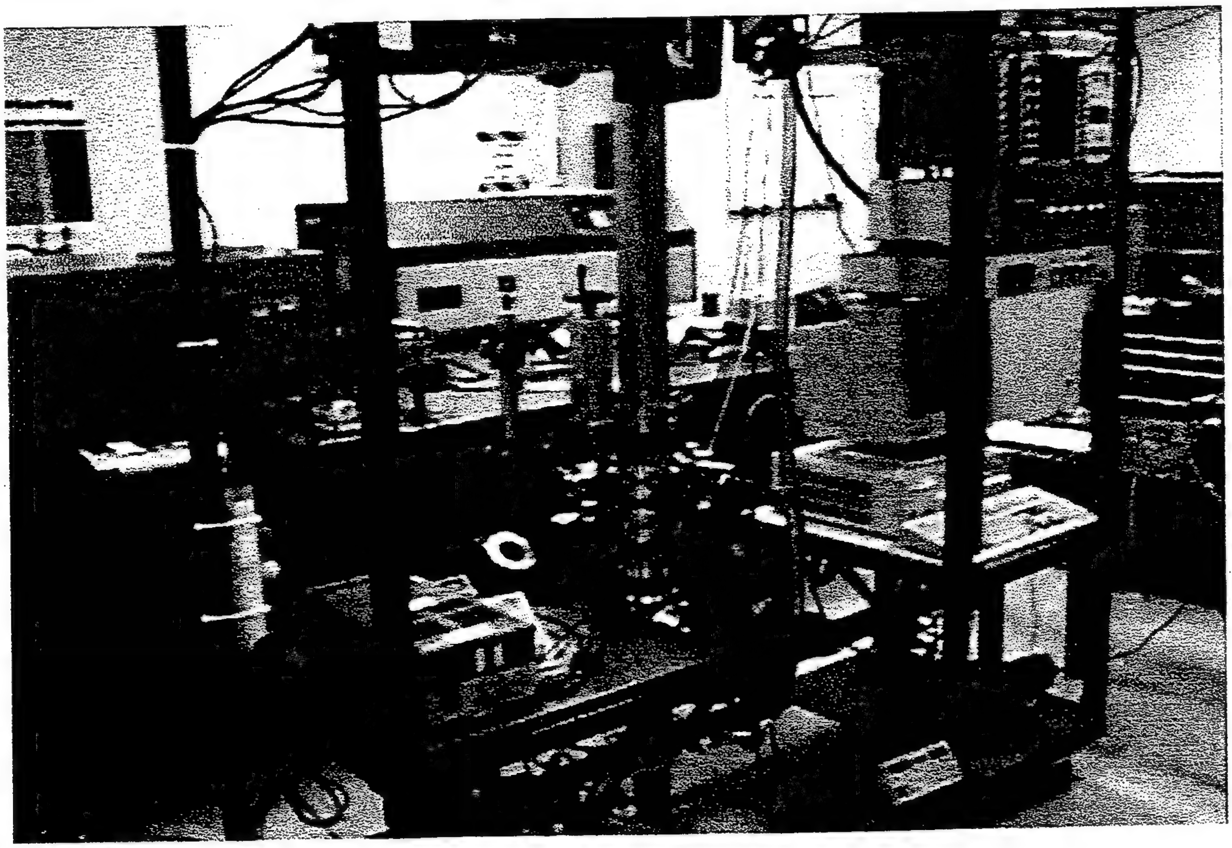
4. Ions are accelerated into TOF and detected by microchannel plate detector.

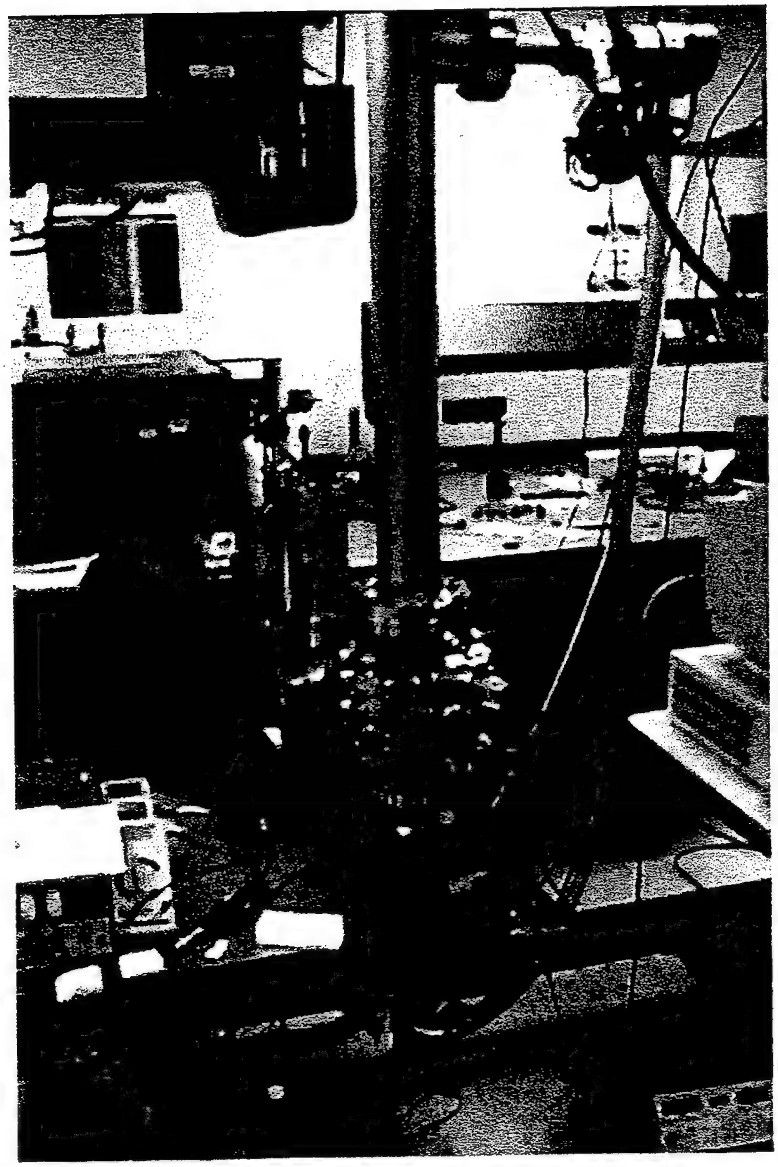
5. Laser scatter onto photodiode gives t_o → OSCILLOSCOPE TRIGGER

6. TOF signal recorded with oscilloscope, transferred to PC, and converted to mass spectrum.

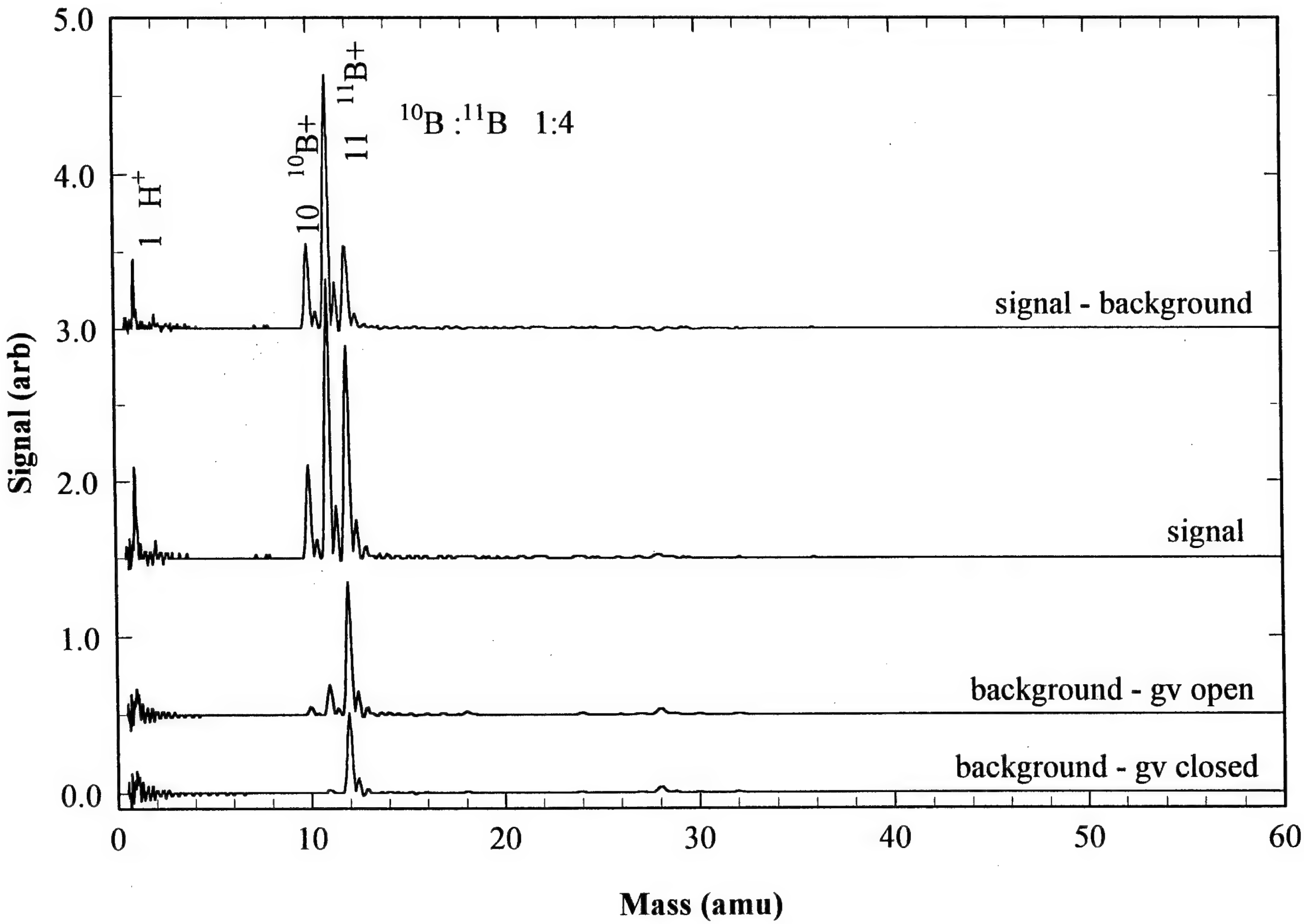
Ionic mass vs. Time-of-Flight





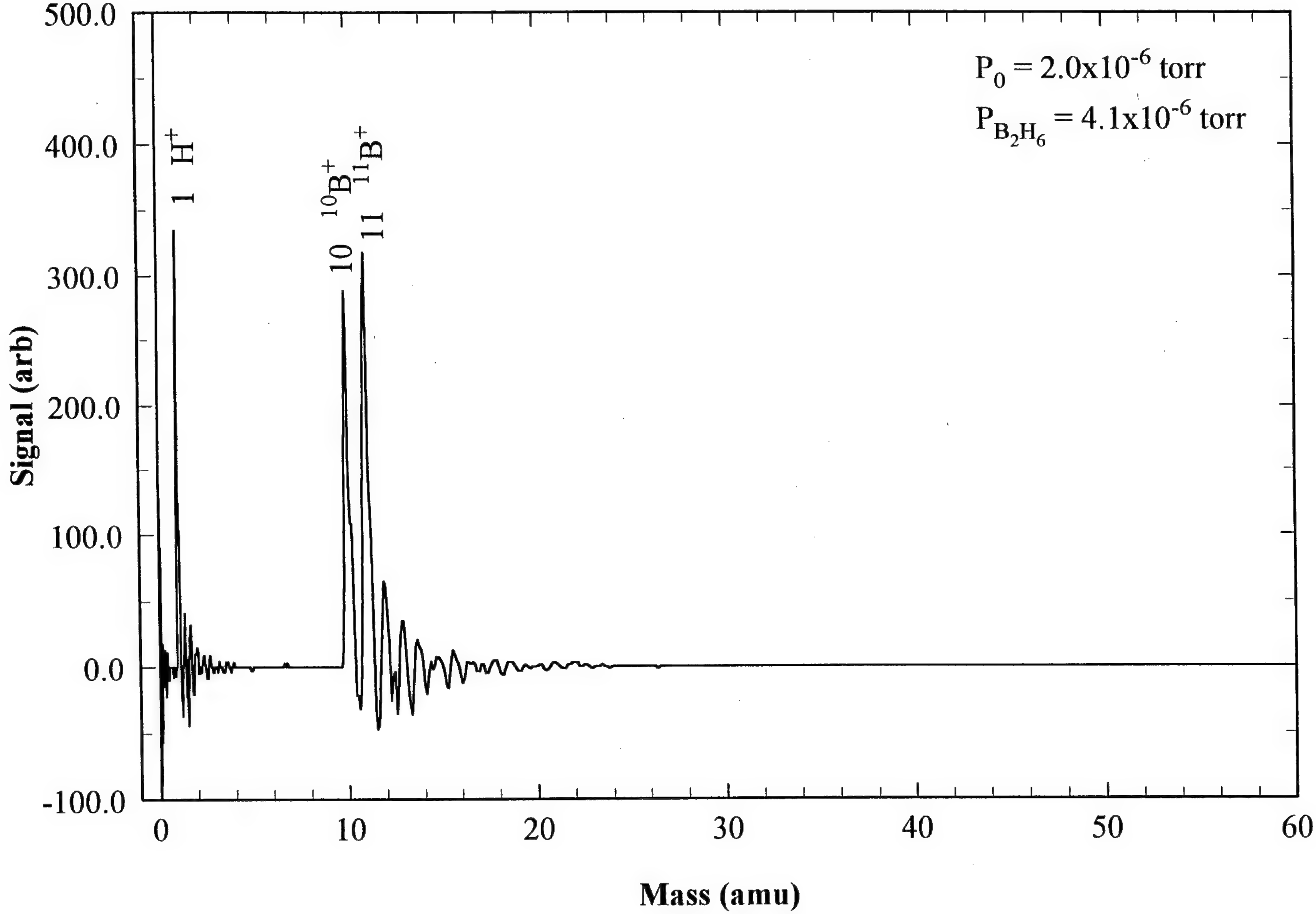


Boron tungsten filament source, 193 nm



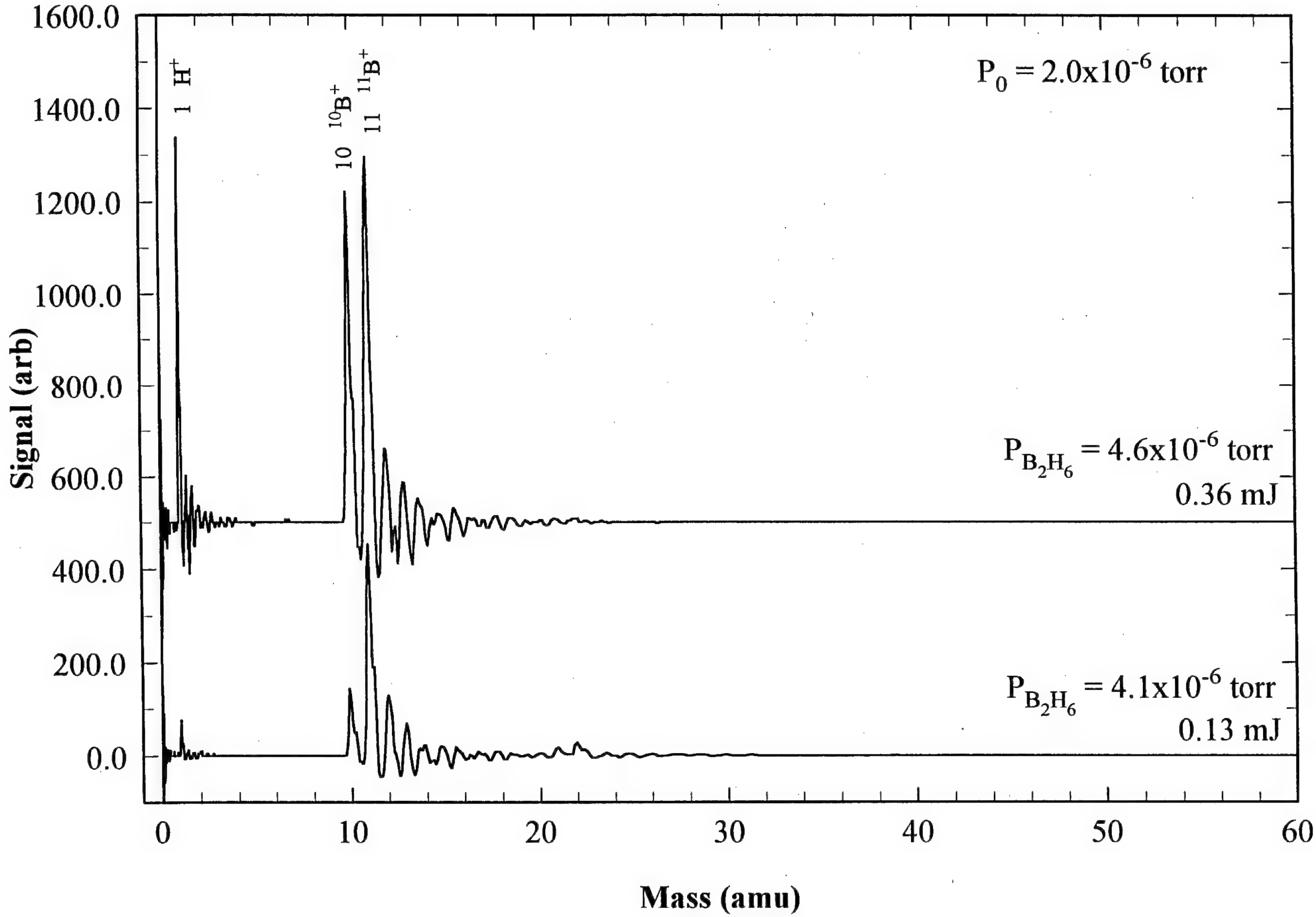
MD213H

B_2H_6, 193 nm



MD230BB

B_2H_6 ionized with different intensities of 193 nm light



MD230J, MD230BB

Multi-photon Ionization and Photofragmentation

Ideally for Time-Of-Flight Mass Spectrometry analysis:

$$XYZ + n\,h\nu \rightarrow XYZ^{+} + e^{-}$$

In practice:

$$XYZ + n\,h\nu \rightarrow \begin{array}{l} XYZ^{+} \\ XY^{+} \\ XZ^{+} \\ YZ^{+} \\ X^{+} \\ Y^{+} \\ Z^{+} \end{array}$$

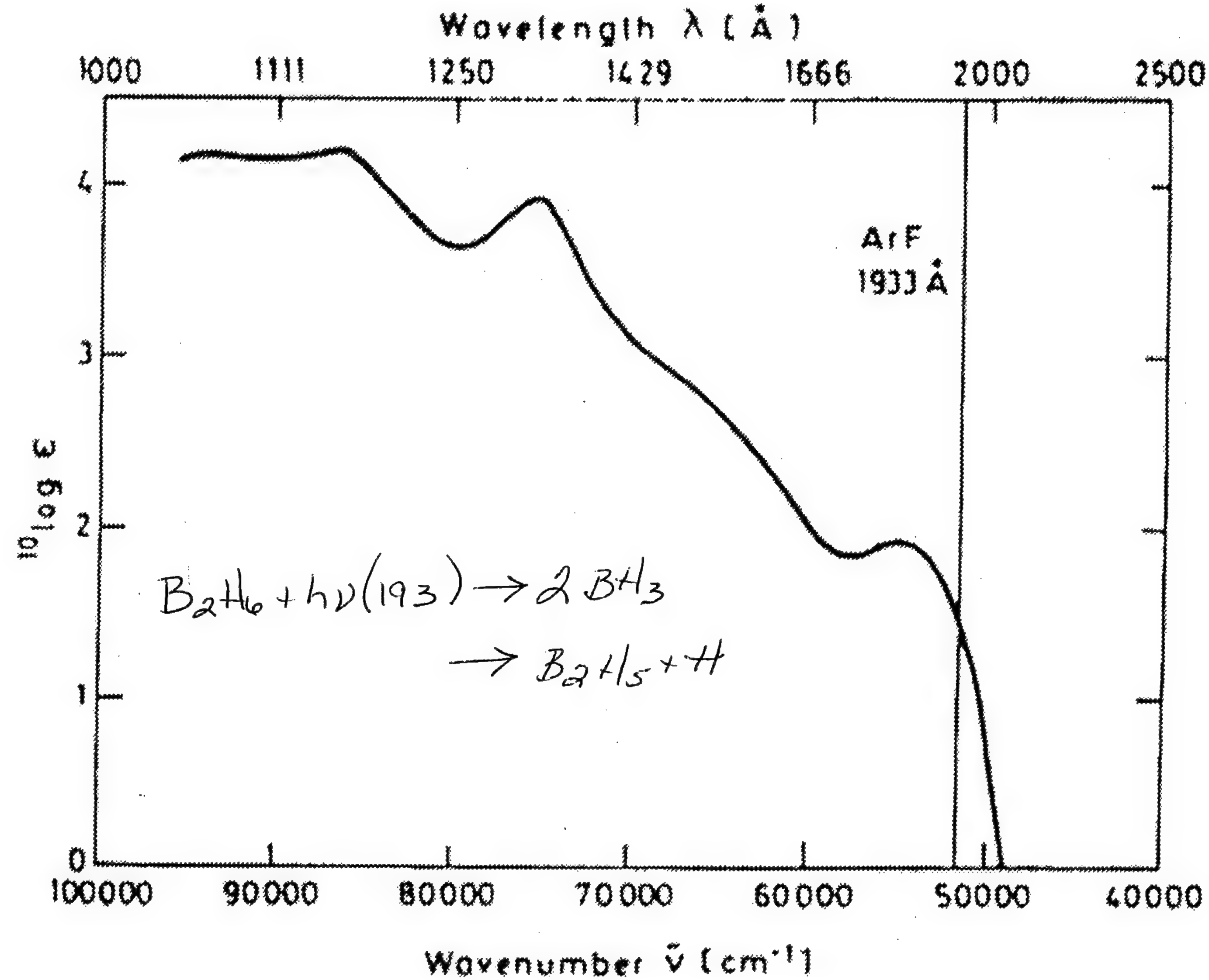
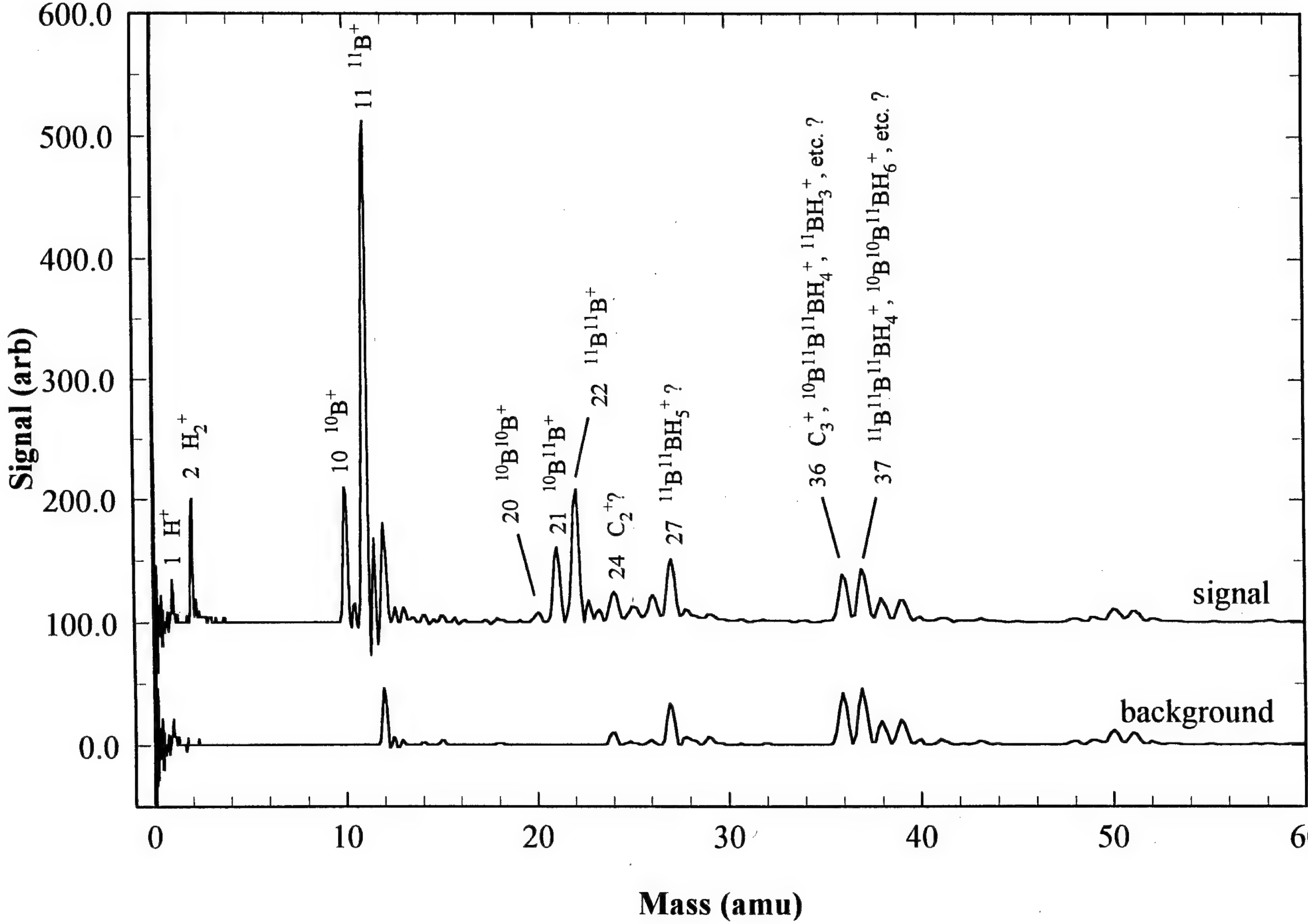


FIG. 1. The VUV absorption spectrum of B_2H_6 in the gas phase after W. Fuß[11]

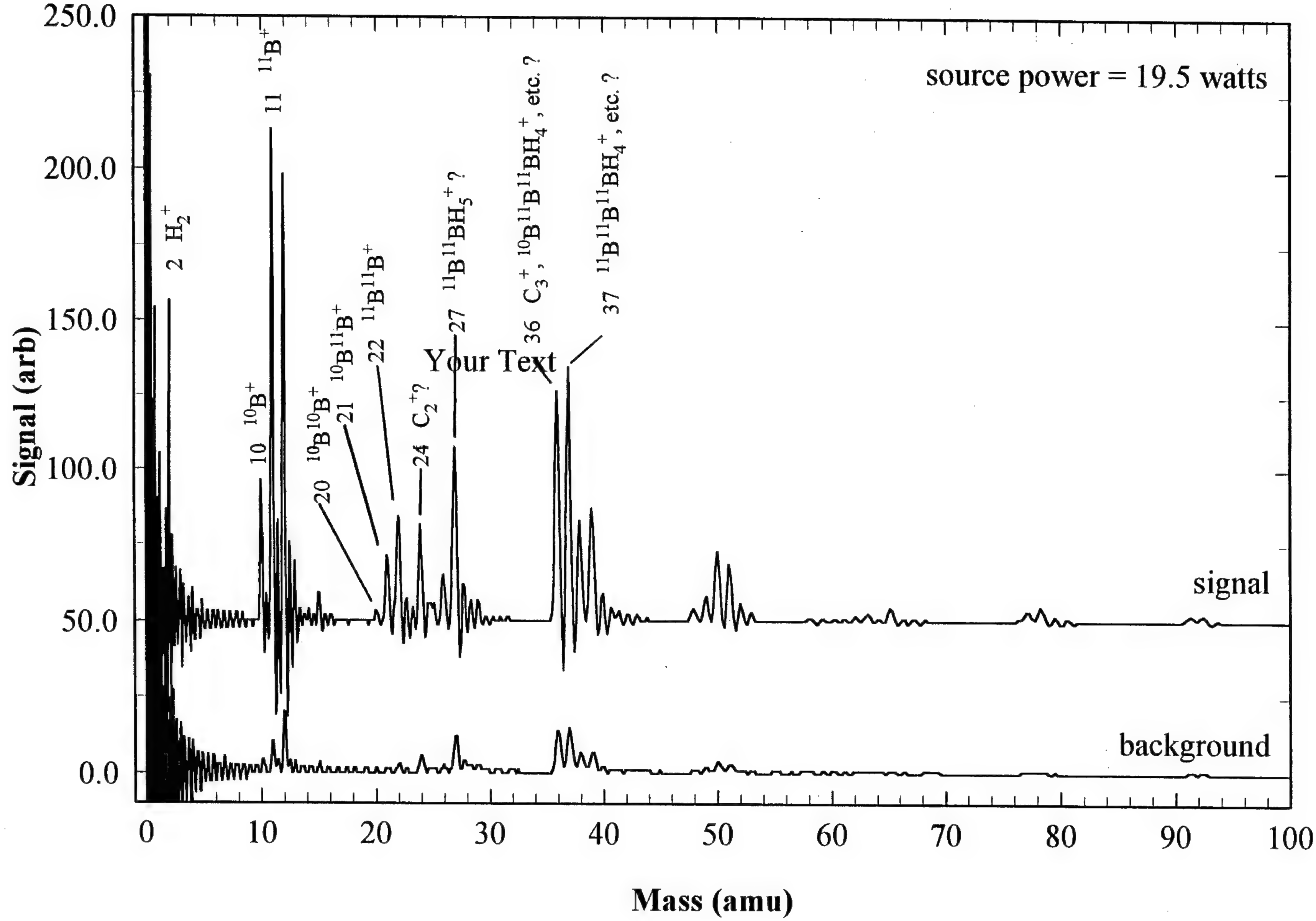
$$\left[\epsilon = \frac{1}{c \cdot 1}\,{}^{10}\log\left(\frac{I_0}{I}\right)\,(1\ \mathrm{mol}^{-1}\ \mathrm{cm}^{-1})\right].$$

B_2H_6, 248 nm

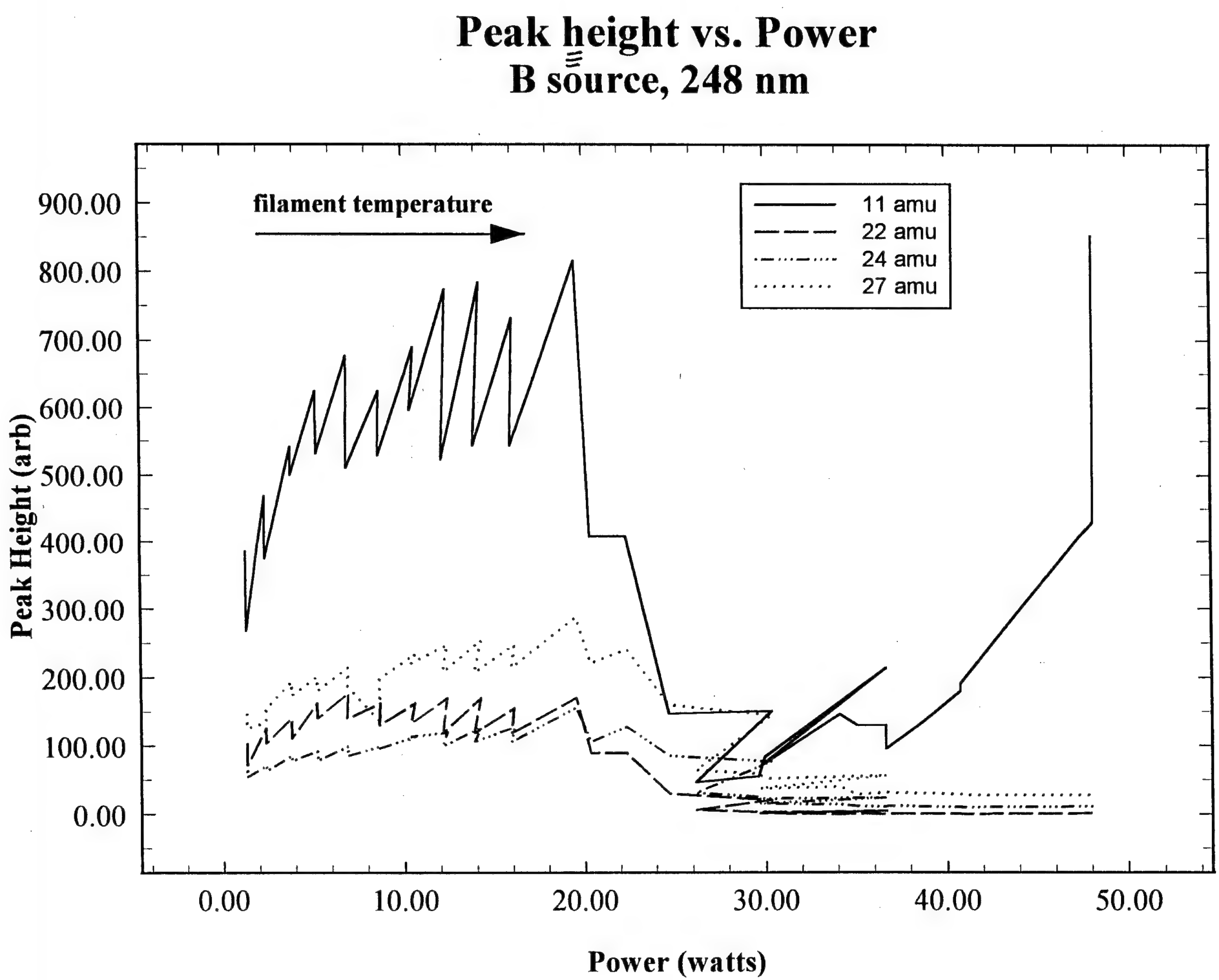


MD257G

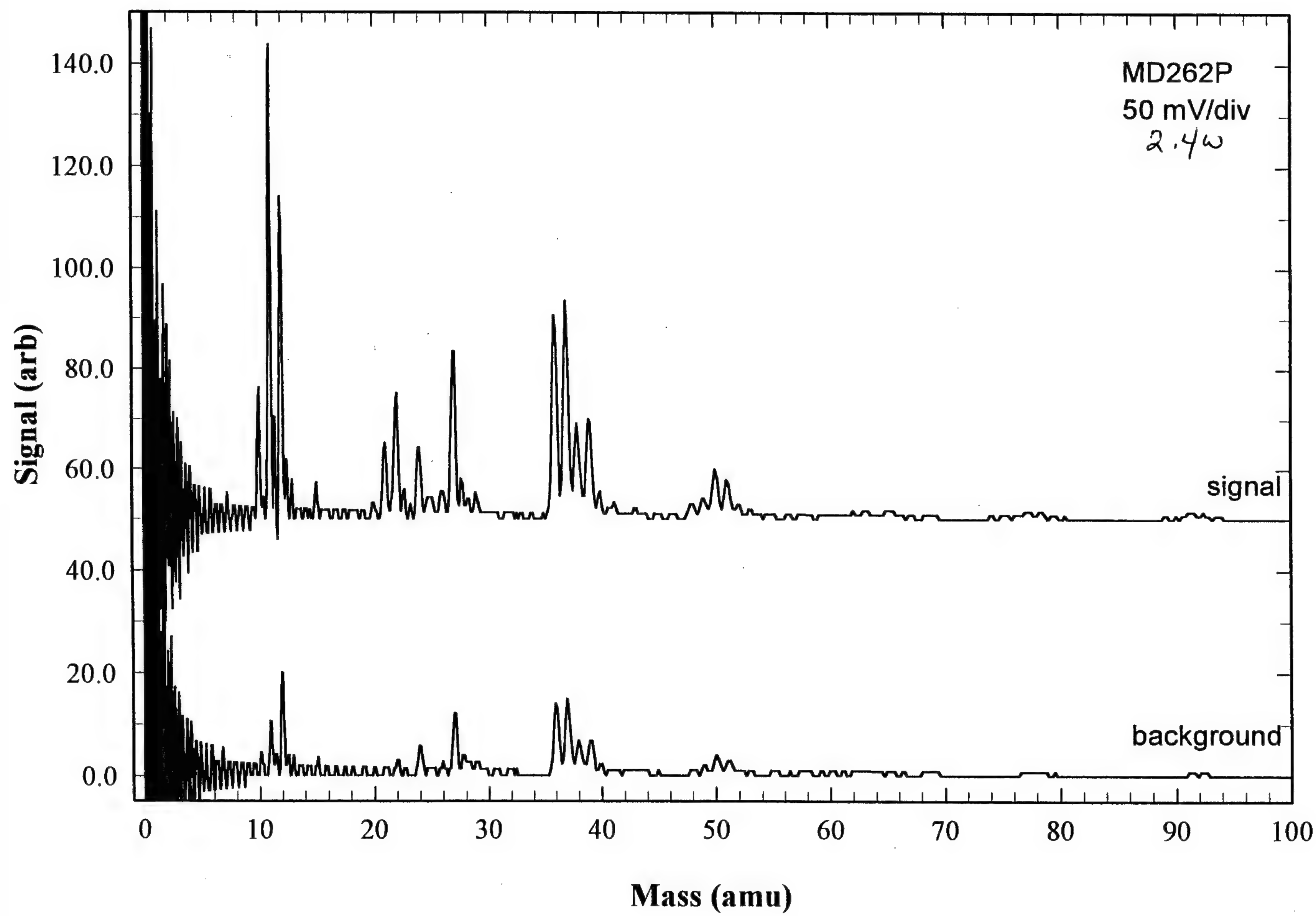
B source, 248 nm

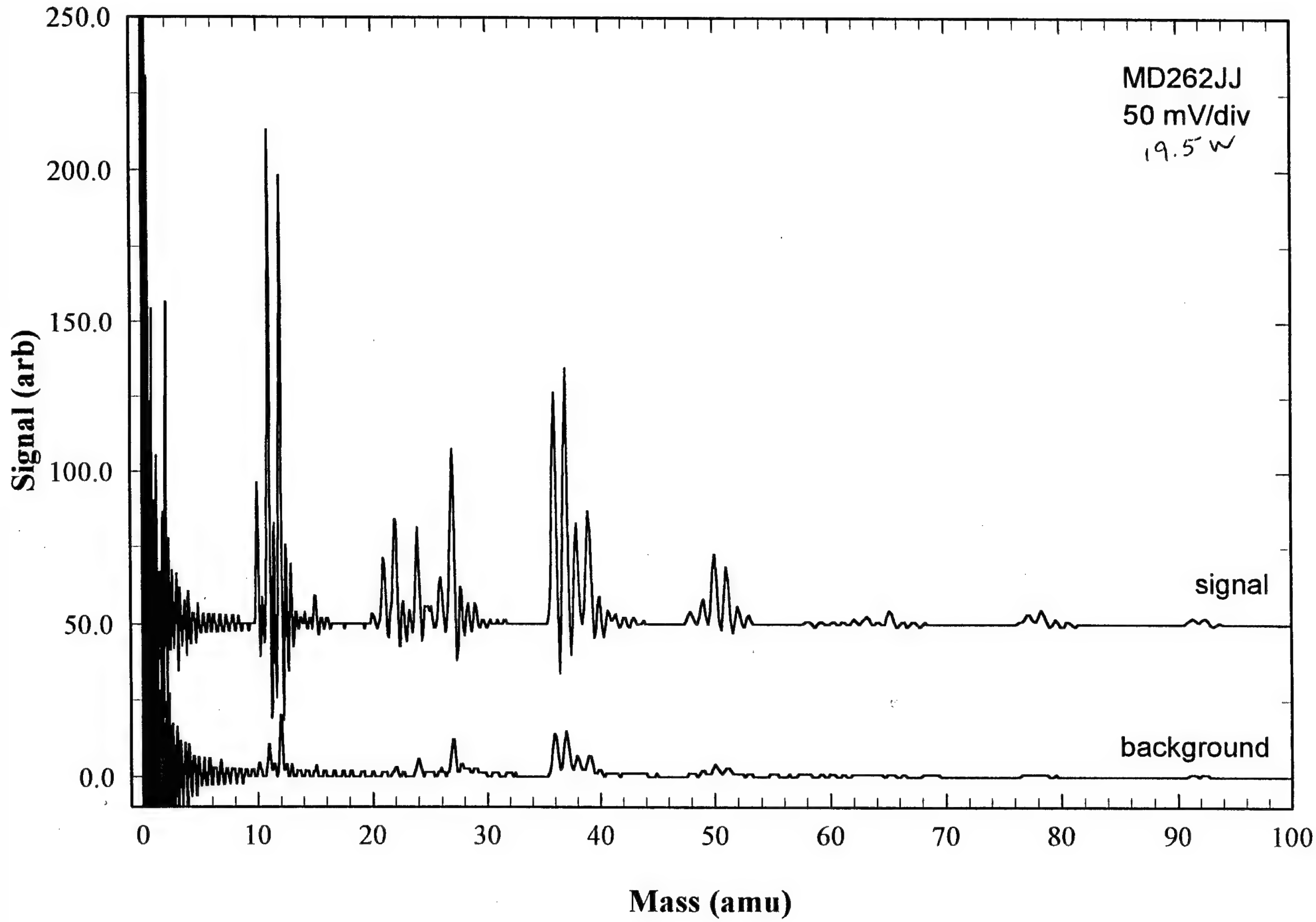


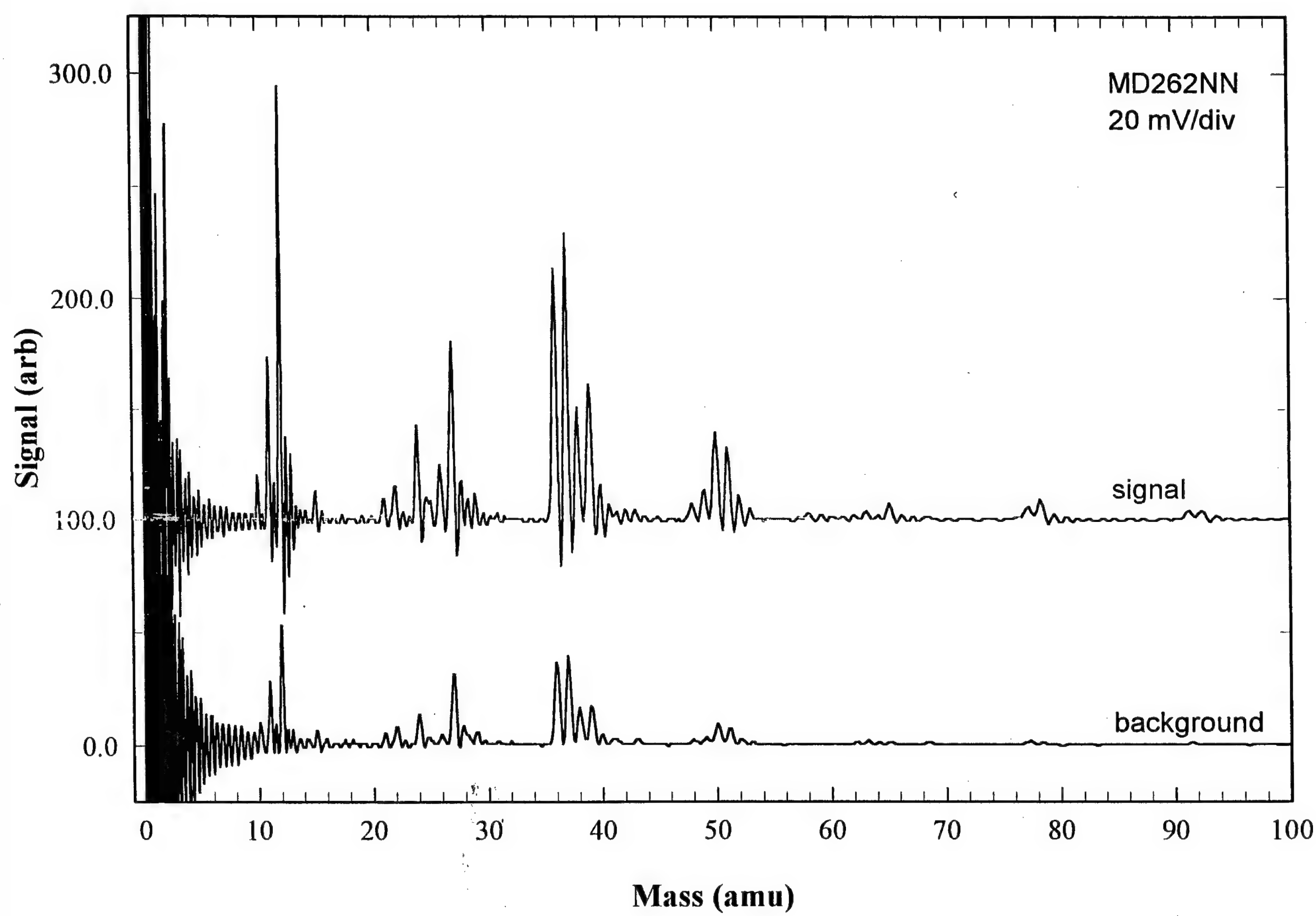
MD262JJ



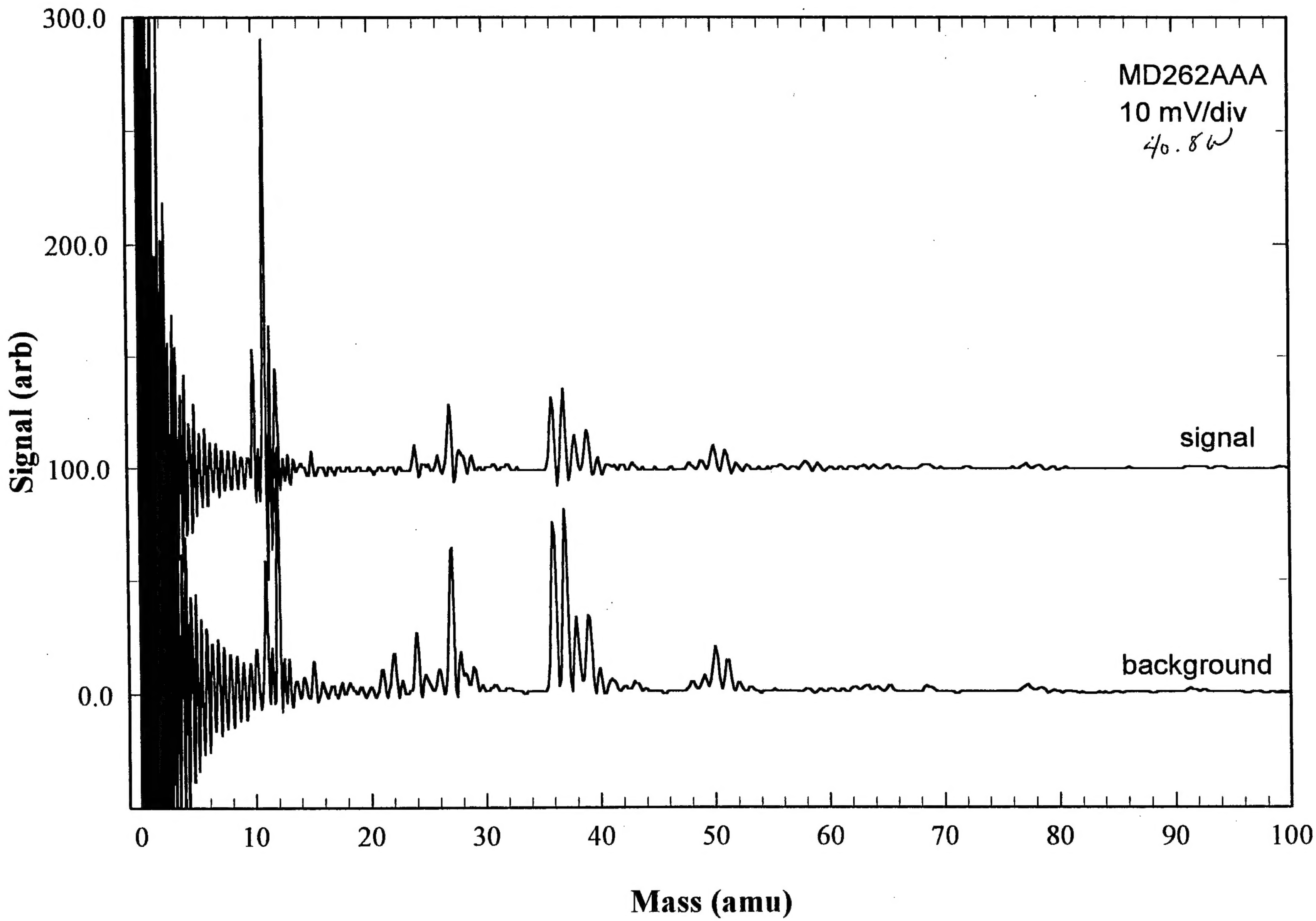
Peak height vs. Power
B source, 248 nm
filament temperature
11 amu
22 amu
24 amu
27 amu
900.00
800.00
700.00
600.00
500.00
400.00
300.00
200.00
100.00
0.00
Peak Height (arb)
0.00
10.00
20.00
30.00
40.00
50.00
Power (watts)

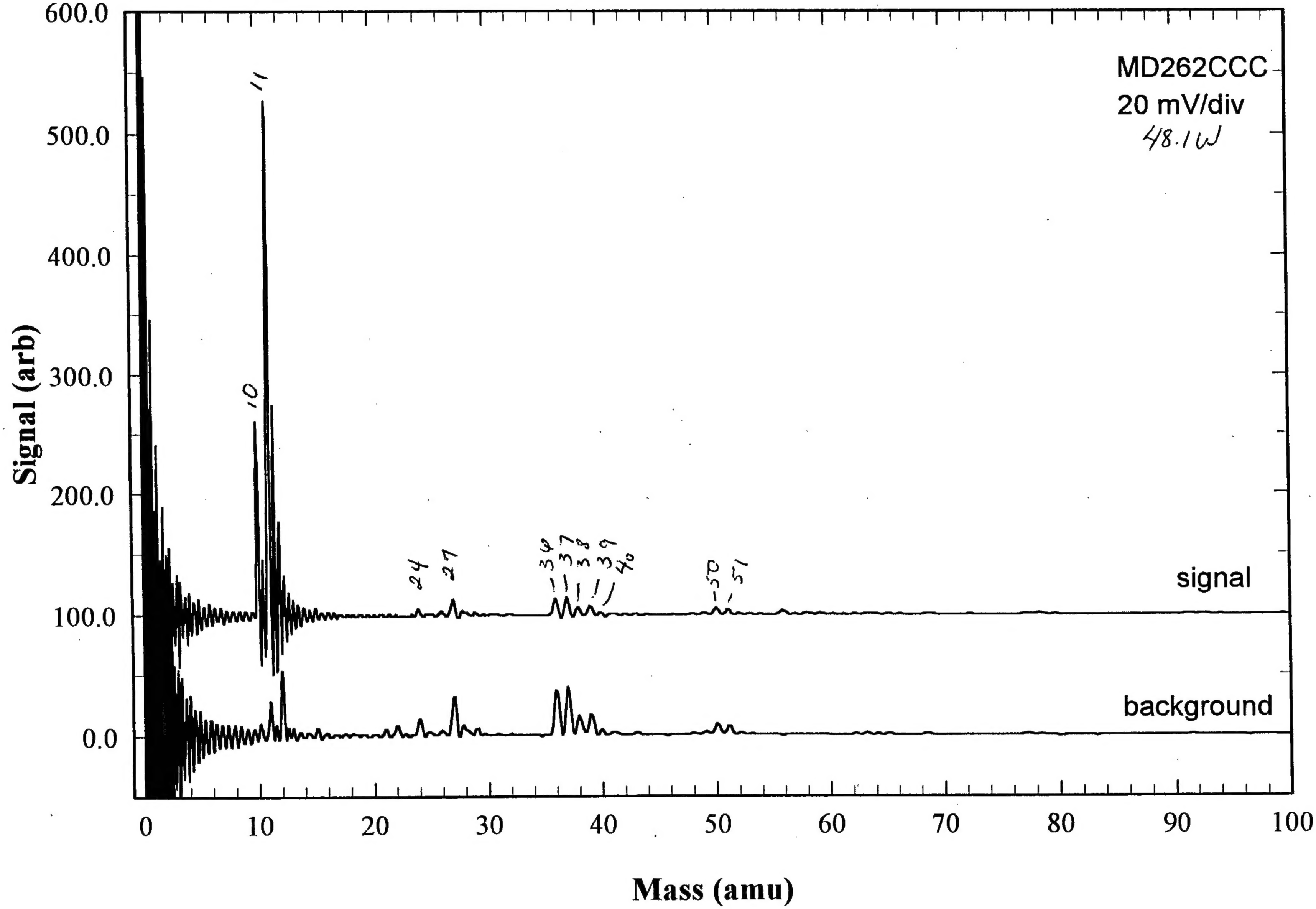

MD262P
50 mV/div
2.4w
signal
background
Signal (arb)
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0.0
Mass (amu)
0
10
20
30
40
50
60
70
80
90
100


MD262JJ
50 mV/div
19.5 W
signal
background
Signal (arb)
Mass (amu)
250.0
200.0
150.0
100.0
50.0
0.0
0
10
20
30
40
50
60
70
80
90
100



MD262NN
20 mV/div
signal
background
Signal (arb)
300.0
200.0
100.0
0.0
Mass (amu)
0
10
20
30
40
50
60
70
80
90
100


MD262AAA
10 mV/div
40.8W
signal
background
Signal (arb)
300.0
200.0
100.0
0.0
Mass (amu)
0
10
20
30
40
50
60
70
80
90
100


MD262CCC
20 mV/div
48.1W
signal
background
Signal (arb)
600.0
500.0
400.0
300.0
200.0
100.0
0.0
Mass (amu)
0
10
20
30
40
50
60
70
80
90
100
11
10
24
27
37
38
39
40
50
51

Summary

- **A system to characterize HEDM sources by time-of-flight mass spectrometry has been assembled, calibrated, and tested. Data show that this system is successful in identifying the products of a HEDM dopant source.**

- Sources for species such as B, and metals such as Al, In, and Ga can easily be made by coating a tungsten filament.

- Varying the intensity of the laser beam is not a satisfactory way to avoid photofragmentation.

- Varying the wavelength of the laser beam shows much more potential for determining the identity of the parent ion.

- Analysis of boron source data indicates that B atoms detected at low filament temperatures are produced by the photofragmentation of boranes. However, as the filament is heated to higher temperatures, B atoms are emitted directly from the source.

Future Directions

- Refine detection and ionization scheme:

  - fix electrical ringing in TOF
  - more experiments with different ionization wavelengths

- Study other sources besides tungsten filament sources

Ultimate goal: be able to take a source off the shelf, quickly characterize and/or verify its ouput, and do a deposition